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IS 12074 (1987): Method for determination of lead by atomic absorption spectrophotometry [CHD 1: Inorganic Chemicals]



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Indian Standard

METHOD FOR
DETERMINATION OF LEAD BY ATOMIC
ABSORPTION SPECTROPHOTOMETER

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

METHOD FOR DETERMINATION OF LEAD BY ATOMIC ABSORPTION SPECTROPHOTOMETER

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Indian Standard

METHOD FOR DETERMINATION OF LEAD BY ATOMIC ABSORPTION SPECTROPHOTOMETER

0. FOREWORD

0.1 This Indian Standard was adopted by the Bureau of Indian Standards on 28 May 1987, after the draft finalized by the Chemical Standards Sectional Committee had been approved by the Chemical Division Council.

0.2 The atomic absorption spectrophotometric method is dependent on the fact that metallic atoms present in ground state will absorb incident radiation from the source, normally hollow cathode lamp, when excited. When radiation from the given excited element is passed through the flame containing ground state atoms of that element, the intensity of the transmitted radiation will decrease in proportion to the amount of the ground state atoms present in flame. A hollow cathode lamp whose cathode is made of the element to be determined provides the radiation. The metal atoms to be measured are placed in the beam of radiation by aspirating the sample solution into an oxidant fuel flame. A monochromator isolates the characteristic radiation from the hollow cathode lamp and a photosensitive device measures the attenuated transmitted radiation. Various metallic elements including lead can be precisely analysed by this technique.

0.3 Various products where lead estimation may be required, include metals and alloys, food products, edible oils, drugs, chemicals, paints, rubber products, water and effluents, petroleum products, etc.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the method for determination of lead by atomic absorption spectrophotometer.

*Rules for rounding off numerical values (*revised*).

2. OUTLINE OF THE METHOD

2.1 The sample is brought into the solution by suitable treatment with acids or acid combinations, diluted with distilled water, filtered and suitable dilutions are made for aspiration into the air acetylene flame. The standard solution is made in the same way for calibration. The most sensitive lead line is 217.0 nm, however, other lines suitable for higher concentration can also be used.

3. INTERFERENCE

3.1 No cationic interference have been reported for the air acetylene flame. A number of anionic interferences have been reported. Phosphates, carbonates, iodides, fluorides and acetates suppress lead absorbance significantly at concentrations ten times greater than lead. These interferences can be largely overcome by addition of EDTA solution so as to give 0.1 molar concentration of EDTA in the final solution for aspiration.

3.2 At 217.0 nm wavelength, non-atomic species in the flame absorb strongly. Where the sample has a high concentration of dissolved solids, it is necessary to use background correction with the help of hydrogen continuum lamp.

4. APPARATUS

4.1 Atomic absorption spectrophotometer provided with background corrector and having following parameters:

- a) Lamp current — depending on the lamp and instrument used
- b) Support — air
- c) Fuel — acetylene
- d) Flame stoichiometry — oxidising
- e) Wavelength and working range

Wavelength (nm)	Band Pass (nm)	Working Range $\mu\text{g/ml}$
217.0	1.0	5 to 20
283.3	0.2	10 „ 40
261.4	0.2	200 „ 800
202.2	0.2	250 „ 1 000
205.3	0.2	2 000 „ 8 000

NOTE — In case of multi-element hollow cathode lamp containing copper, the second sensitive line for lead, that is, 283.3 nm may be taken to avoid the interference of copper.

5. REAGENTS

5.1 Pure Lead Metal — 99.99 percent.

5.2 Concentrated Nitric Acid — see IS : 264-1976*.

5.3 Concentrated Hydrochloric Acid — see IS : 265-1976†.

5.4 Standard Lead Solution — Dissolve 1.0 g of lead in 1 : 1 nitric acid, dilute to one litre with distilled water to give 1 000 µg/ml of lead.

6. SAMPLE PREPARATION

6.1 Metals and Alloys — A suitable quantity of sample is dissolved in hydrochloric acid or a mixture of hydrochloric acid and nitric acid, evaporated to dryness, again dissolved in hydrochloric acid, diluted, filtered and made up to known volume. A suitable dilution is made for determination of lead before aspirating in the flame. Concentration of solution by ion exchange or by solvent extraction should be done where lead is expected to be present in very low quantities.

6.2 Water and Effluents — Lead content in water is usually at very low level. Concentration technique is required before feeding to flame. Concentration may be done by evaporation of solvent extraction or by ion exchange method.

6.3 Minerals and Ores — Finely divided particles are dissolved in hydrochloric-nitric acid mixture and evaporated to dryness. It is again taken in hydrochloric acid, diluted and filtered to remove siliceous matter.

Residue is dried, ignited and fused with fusion mixture. Extract in hydrochloric acid is taken and mixed with original solution. The final solution is made up to known volume.

6.4 Food Products, Clinical Samples, Organic Materials, Paints, etc — The sample is brought into solution by wet digestion with nitric acid/perchloric acid or dry ashing at 450°C and dissolving in nitric acid. Filtrate is made up to suitable known volume.

6.5 Atmospheric Particulates — Membrane filter containing particulates is leached in nitric acid and filtered. The solution is made to a known volume with distilled water.

*Specification for nitric acid (*second revision*).

†Specification for hydrochloric acid (*second revision*).

6.6 Coal and Coke — After ashing at 550°C, it is dissolved in *aqua-regia*, filtered and diluted to known volume.

7. PROCEDURE

7.1 Optimize the response of instrument by adjustment of burner height and flame. Aspirate water to get zero absorption, when stable response is observed, aspirate standards (at least 4) and note down absorption.

Aspirate sample to get absorption of the sample. Prepare calibration curve by plotting the net absorption value of the standard against concentration in µg/ml of lead. Locate the point of the sample absorption and calculate the concentration of lead in the sample.

8. CALCULATION

$$\text{8.1 Lead percent by mass} = \frac{C \times V}{10^6} \times \frac{100}{M}$$

where

C = concentration of lead in µg/ml in final solution,

V = volume in ml of final solution, and

M = mass in g of the sample in final solution.